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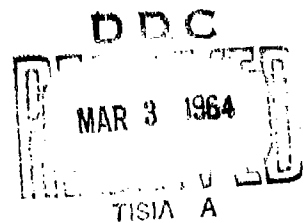
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SEALS FOR 12,500 PSIG AIR SYSTEMS
SECOND PROGRESS REPORT

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RUBBER LABORATORY
MARE ISLAND NAVAL SHIPYARD



TECHNICAL REPORT

430727

64-9

SEALS FOR 12,500 PSIG AIR SYSTEMS
SECOND PROGRESS REPORT

REPORT NO. 28-12

Project No. S-F013-13-01

Task No. 907

Identification No. 907-7

RUBBER LABORATORY
MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

Prepared 19 February 1964

ABSTRACT

The ability of Viton B O-rings of 90 Shore A hardness to seal 12,500 psig air pressure was evaluated under dynamic and static simulated-service conditions using solid Teflon back-up rings. Both dynamic and static seals showed small volume leakage after total elapsed times under pressure ranging from 35 to 58 hours.

The O-rings showed no defects or evidence of extrusion after test. Leakage may have been due to slow recovery of the O-rings from compression, microscopic defects in the metal and/or rubber surfaces, or to the presence of small particles of compressed Molykote powder, the lubricant used on the O-rings. The rate of leakage was much less than previously reported for similar tests using spiral Teflon back up rings.

The swelling of the O-rings caused by sudden release of pressure was low, for example: 1.2% after 1,000 psig pressure and 3.6% after 12,500 psig pressure. The measurements were made one hour after pressure release.

The physical properties of Viton B O-rings were not greatly affected by six months exposure to oxygen at 60°F and 1,750 pressure.

For future work, it is proposed to investigate the sealing abilities of dual O-rings, of O-rings made of nitrile rubber, and of O-rings lubricated with Kel F grease.

REFERENCES

- (a) BUSHIPS ltr FO13 13 01, Ser 634C1-470 of 7 Jun 1963
- (b) BUSHIPS ltr FO13 13 01, Ser 634C1-692 of 14 Jul 1961
- (c) BUSHIPS ltr FO13 13 01, Ser 634C1-499 of 24 May 1962
- (d) BUSHIPS ltr FO13 13 01, Ser 634C1-846 of 5 Sep 1962
- (e) Military Specification MIL-G-23652(SHIPS) of 28 Mar 1963; "Gasket and Packing Material Petroleum and Phosphate Ester Fluid Resistant"
- (f) NAVSHIPYD MARE Rubber Laboratory Report 28-11 of 26 Jun 1963; "Seals for 12,500 psig Air Systems. First Progress Report"
- (g) NAVSHIPYD MARE Rubber Laboratory Report 28-10 of 10 July 1962; "Seals for 5200 psi Air Systems. Final Report"
- (h) BUSHIPS Instruction 9230.15B Ser 648F-2475; Ser 648F-2475 of 27 Dec 1962

INTRODUCTION

1. The Rubber Laboratory was authorized by reference (a) to continue work on the development of seals for 12,500 psig air service as outlined in reference (b) and modified by references (c) and (d). The work assigned included the development of a specification for the O-rings which would parallel specification MIL-G-23652, reference (e).

2. This is the second progress report of this investigation. The first progress report, Report 28-11, reference (f), described the results of simulated service tests at 12,500 psig gas pressure of Viton B O-rings of 90 Shore A hardness using spiral Teflon back-up rings. Three test devices, each containing two dynamic and two static O-ring seals, were used. The static seals in the three test devices leaked badly after 31, 38, and 93 cycles, respectively. Each cycle consists of at least one-half hour under pressure followed in sequence by rapid release of pressure, movement of the piston with the dynamic seals, and re-pressuring. This report describes similar tests of 90 Shore A O-ring seals using solid Teflon back-up rings instead of spiral back-up rings. Also included herein is a study of the effect of sudden release of air pressure on the swelling of Viton B O-rings and a study of the effects of prolonged exposure to oxygen at 1,500 psig pressure on the tensile properties of Viton B O-rings.

FORMULATION USED FOR O-RINGS

3. The O-rings tested were molded from Rubber Laboratory stock 377-112. This was a Viton B stock meeting the requirements of specification MIL-G-23652, Type II. This specification covers packings and gaskets to be used in 5,200 psig air systems. Stock 377-112 was developed for this service, as described in Report 28-10, reference (g). The formulation of the stock and the cure employed in manufacturing the O-rings are given below:

Stock 377-112

| | |
|----------------------|-----|
| Viton B | 100 |
| Thermax | 10 |
| Philblack E | 15 |
| Maglite D | 15 |
| Stearic acid | 0.2 |
| Di-isooctyl sebacate | 3 |
| Diak No. 3 | 4 |

Cure: 30 min at 300°F in press, plus 1 hr each at 200°, 250°, 300°, 350°, and 400°F in oven, plus 24 hrs at 450°F in oven.

TESTING PROCEDURES

Sealing Tests

4. The device used for testing the ability of the O-rings to seal 12,500 psig air pressure is shown in Appendix 1. It consisted of a hollow cylinder, a cap for each end of the cylinder, and a piston designed to slide inside the cylinder. All parts were made of corrosion-resistant steel. The cylinder had a circumferential groove at each end in which the O-rings for static testing were installed. The piston had a circumferential groove at each end in which the O-rings for dynamic

Testing were installed. All grooves were wide enough for Teflon back-up rings as well as O-rings. The back-up rings were installed on the low pressure side of the O-rings to prevent the O-rings from being extruded through the downstream clearance. To permit the use of solid Teflon back-up rings, the upstream side of each O-ring groove was a removable ring or disk, as shown in Appendix 1.

5. The O-rings on the cylinder were AN6230-7 size with a cross-sectional diameter of $0.139 \pm .004$ inch and an internal diameter of $2.359 \pm .010$ inches. The O-rings on the piston were AN6227-27 size with a cross-sectional diameter of $0.139 \pm .004$ inch and an internal diameter of $1.484 \pm .006$ inches. The depths of the grooves in the cylinder and piston were designed to yield 8% compression of O-rings having the minimum allowable cross-sectional diameter. The measured diametric clearance was 3 to 5 mils between the cylinder and the end caps, and between the piston and the cylinder.

6. The apparatus was operated by applying air at 12,500 psig pressure to the ends of the cylinder through the holes in the caps. The dynamic and static seals were thus pressurized simultaneously. Movement of the piston was effected by establishing a pressure differential between the two ends. The 12,500 psig air pressure was obtained by means of a diaphragm-type compressor, Model 3033 manufactured by the Pressure Products Industries, Hattboro, Pennsylvania.

7. Three test devices were used. Before assembling each device, the O-rings to be tested and the walls of the cylinders were wiped with Molykote, a powdered molybdenum disulfide lubricant manufactured by the Alpha Corporation, Greenwich, Connecticut. The O-rings and solid Teflon back-up rings were installed in the grooves with the back-up rings on the low pressure side of the corresponding O-rings, as shown in Appendix 1.

8. Solid Teflon back-up rings were used instead of spiral Teflon back-up rings because the results of the tests described in Report 28-11 indicated that the observed leakage of the Viton B O-rings may have been due to the use of spiral back-up rings. Examination of the O-rings after the earlier tests revealed that the only discontinuity on the surface of each O-ring was a permanent distortion in the area which contacted the spiral back-up ring at, and between, the ends of the spiral. This distortion may have reduced the compression of the O-ring at this point sufficiently to cause leakage. The use of a solid back-up ring would eliminate this condition.

9. The nominal dimensions of the solid Teflon back-up rings were as follows:

| <u>O-ring Size</u> | <u>Back-up Ring Dimensions</u> | | |
|--------------------|--------------------------------|------------------------|--------------|
| | <u>Thickness</u> | <u>Inside Diameter</u> | <u>Width</u> |
| AN6230-7 | 0.050 inch | 2-13/32 inches | 0.120 inch |
| AN6227-27 | 0.050 inch | 1-17/32 inches | 0.120 inch |

10. The three devices were assembled and connected to the compressor. The devices were immersed in water so that any leaks would be readily apparent.

11. The sequence of pressure application and release is given below. Steps "a" through "d" were considered to be one cycle.

a. Air at 12,500 psig was applied first to one end of each test device and then immediately to the other end. These ends are identified hereinafter as A and B respectively. The piston was initially at B, being forced there by the pressure at A.

b. After 30 minutes, B and A were exhausted in quick succession. The piston remained at B. Approximately 7 seconds were required to exhaust B and 20 seconds were required to exhaust A.

c. After 15 seconds, 12,500 psig air pressure was applied to A and then to B. The piston remained at B.

d. A was exhausted to allow the piston to be forced to A by the 12,500 psig pressure in B. A was then re-pressurized. The piston remained at A.

e. After 30 minutes the procedures in paragraphs b, c, and d were repeated except that the piston was initially at A and was moved to B.

f. On subsequent cycles, the procedures were alternated in order to move the piston back and forth.

g. The test was continued for eight hours per day, five days per week until failure occurred as evidenced by continuously escaping gas. The pressure was left on overnight and weekends, deteriorating from 12,500 psig to a minimum of about 5,000 psig. A minimum of 200 cycles without failure was arbitrarily established as the requirement for satisfactory O-rings.

Swelling Tests

12. The swelling of O-rings, size AN6230-7, made from stock 377-112, was measured after quick release of air pressure. The conditions of the test were as follows:

- a. Time of exposure to air pressure: 7 days.
- b. Temperature of air: between 70° and 80°F.
- c. Air pressures used in successive tests on different O-rings: 1,000; 2,000; 3,000; 5,000; 7,000; and 12,500 psig.
- d. Elapsed time for pressure to reach 0 psig after release: between 1 and 2 minutes.
- e. Elapsed time between pressure release and measurement of volume of O-rings: 1 hour.

Oxygen Aging Tests

13. In order to obtain a measure of the effect of continuous exposure of the O-rings to 12,500 psig air, O-rings of size AN6230-7 made of stock 377-112 were aged for six months at an average ambient temperature of 60°F in oxygen at 1,750 psig pressure. Tensile strength, ultimate elongation and modulus at 100% elongation were measured in accordance with Methods 4111, 4121, and 4131 of Federal Test Method Standard No. 601, respectively, on six unaged O-rings and six oxygen-aged O-rings. At the end of the aging period, the oxygen pressure was released slowly, approximately one hour being required to reduce the pressure from 1,750 psig to 0 psig. The volume of the O-rings was measured before and after the oxygen aging. The aged volume was measured one hour after removal from the oxygen.

RESULTS

Sealing Tests

14. Continuous leakage from the dynamic seals of one test device was observed after 31 cycles and from the static seals of the same test device after 42 cycles. No leakage was observed from the other two devices at this time. Examination of the O-rings which leaked revealed a few particles of metal, cloth fibers, and rust imbedded in the surface of the O-rings. These foreign particles may have caused the leakage. No other imperfections were noted in the O-rings or the Teflon back-up rings.

15. Additional filters were installed in the supply lines to prevent foreign particles from entering the test devices. The test device which leaked was reassembled with new O-rings and new solid back-up rings. The testing of the other two test devices was resumed. The reassembled test device with the new rings leaked from both the dynamic and static seals after 69 cycles, and the other two devices

leaked similarly after a total of 115 cycles. The leakage from the dynamic seals of all three devices was of small volume and continuous during the first 1/4 to 6 minutes under 12,500 psig pressure, after which the leakage stopped. The static seals of all three devices also leaked slightly as indicated by one large bubble every 2 to 10 minutes while under 12,500 psig pressure. Several cycles were run after leakage was first observed, and approximately the same leakage pattern was repeated during the subsequent cycles.

16. While the observed leakage was not great, it was not desirable. The test devices were disassembled and the O-ring and back-up rings were examined. The O-rings showed no evidence of extrusion or other defects. The back-up rings were in good condition except for a small amount of extrusion into the downstream clearances. The cause of leakage may have been due to slow recovery of the O-rings from compression, microscopic defects in the metal and/or rubber surfaces, or to the presence of small particles of compressed Molykote.

17. The rate of leakage observed was much less than described in Report 28-11 for similar tests wherein spiral rather than solid back-up rings were used, and the total number of cycles without leakage was approximately double. The results indicate that solid back-up rings rather than spiral back-up rings should be used in 12,500 psig air systems. However, this conclusion should be verified, since it is based on relatively few tests.

Swelling Tests

18. The swellings of the O-rings after release from 7 days exposure at 70-80°F to air at various pressures are given below:

Air Pressure, psig Swelling, % *

| | |
|--------|-----|
| 1,000 | 1.2 |
| 2,000 | 1.5 |
| 3,000 | 1.8 |
| 5,000 | 3.5 |
| 7,000 | 3.5 |
| 12,500 | 3.6 |

* Measured one hour after pressure release

19. The data show that the swelling values were quite low, even for 12,500 psig pressure. It is also interesting to note that the swellings of specimens exposed to pressures of 5,000 psi and above were the same.

Oxygen Aging Tests

20. The effect of aging for six months at an average ambient temperature of 60°F in oxygen at 1,750 psig pressure on the physical properties of the O-rings made from stock 377-112 is given below:

| | <u>Initial</u> | <u>After 6 months under 1,750 psig oxygen pressure</u> | <u>% Change</u> |
|---------------------------------|----------------|--|---------------------|
| Tensile strength, psi | 2240 | 1910 | -14.7 |
| Ultimate elongation, % | 130 | 130 | 0.0 |
| Modulus at 100% elongation, psi | 1860 | 1670 | -10.2 |
| Volume swelling, % * | -- | 1.4 | -- |

* Measured one hour after pressure release

21. Although one hour was taken to reduce the oxygen pressure from 1,750 psig to 0 psig at the end of the six month aging period, this rate of pressure drop was not slow enough to permit the dissolved oxygen from diffusing out without forming microscopic pores in the rubber. This fact was evidenced by the 1.4% volume increase observed one hour after pressure release. The microscopic pores formed in the rubber when the pressure was released undoubtedly caused the observed reduction in tensile strength and modulus. In future oxygen-aging tests, the pressure will be reduced at a much slower rate in an effort to eliminate this effect.

22. In spite of the presence of microscopic pores in the rubber, the effect of the oxygen aging on the properties of the O-rings was small. Since aging in oxygen at 1,750 psig pressure is approximately equivalent in terms of oxidation to aging in air at 8,600 psig pressure, these results indicate that the properties of Viton B O-rings would probably not be greatly affected by extended service in 12,500 psig air systems.

CONCLUSIONS

23. The suitability of Viton B seals for use in 12,500 psig air systems is open to question. Further sealing tests should be performed.

24. Viton B stock 377-112 is suitable for the manufacture of seals for high pressure air systems from the standpoint of swell due to dissolved air.

FUTURE WORK

25. The effect of using Kel F Grease No. 90 rather than Molykote powder for lubricating the O-ring will be studied. Kel F Grease No. 90 is listed in BUSHIPS Instruction 9230.15B, reference (h), for use in high pressure air (1,500 psi and above).

26. Sealing tests will be performed on softer (70-Shore A) Viton B O-rings. Softer O-rings will recover more rapidly during cycling and will seal irregularities in the metal surfaces better than the harder O-rings.

27. The use of two O-rings held in adjacent grooves to form the seal at a given location will be investigated. New test devices have been made wherein each piston for testing the dynamic seal has two O-ring grooves instead of one at each end, and each stationary cylinder also has two O-ring grooves instead of one at each end for testing the static seals.

28. The effect of aging in high pressure oxygen on the properties of O-rings for 12,500 psig air systems will be studied further. The aging will be performed at a moderately elevated temperature to accelerate the effect.

29. The suitability for this service of O-rings made of Hycar 1071, a carboxylated butadiene acrylonitrile copolymer, will be determined.

PERSONNEL

Tests conducted by:

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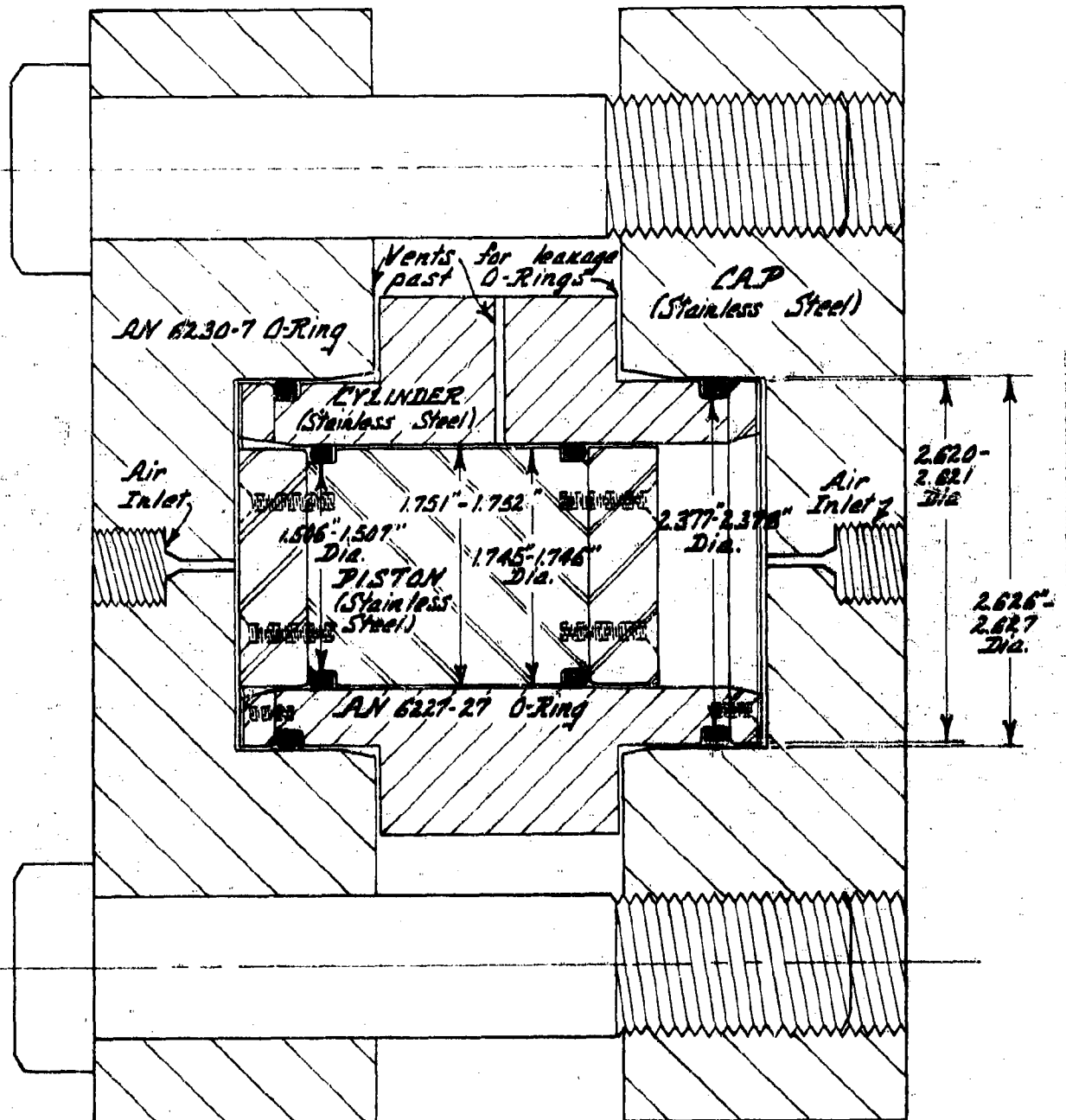
R. E. Morris
R. E. Morris, Head, Rubber Laboratory

APPENDICES

1. Drawing. Cross-sectional view of device for static and dynamic sealing tests of O-rings for high pressure air service.

Distribution of Report

Abstract cards



CROSS-SECTIONAL VIEW OF DEVICE FOR STATIC AND DYNAMIC SEALING
 TESTS OF O-RINGS FOR HIGH PRESSURE AIR SERVICE

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Mare Island Naval Shipyard, Rubber Laboratory Report 28-12 of 19 Feb 1964. SEALS FOR 12,500 PSIG AIR SYSTEMS, SECOND PROGRESS REPORT, by A. E. Barrett. Project No. S-P013-13-01, Task 907, Identification No. 907-7. The ability of Viton B O-rings to seal 12,500 psig air pressure was evaluated under simulated service conditions using solid Teflon back-up rings. Both dynamic and static seals showed small volume leakage after elapsed times under pressure ranging from 35 to 58 hours. The O-rings showed no defects or evidence of extrusion after test. Leakage may have been due to slow recovery of the O-rings from compression, microscopic defects in the metal and/or rubber surfaces, or to the presence of small particles of compressed Molykote powder, the lubricant used on the O-rings. The physical properties of Viton B O-rings were not greatly affected by 6 months exposure to oxygen at 60°F and 1,750 psig pressure. The swelling of Viton B O-rings after release from 7 days exposure to air at 1,000 to 12,500 psig pressure was low, ranging from 1.2 to 3.6 percent.

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